

**Final Report for AOARD Grant 094114 “Towards Corrected and Completed Atomic Site Occupancy Analysis of Superalloys Using Atom Probe Tomography Techniques”**

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**PI and Co-PI information:**

Name of Principal Investigator: Simon Ringer

e-mail address: [simon.ringer@sydney.edu.au](mailto:simon.ringer@sydney.edu.au)

Institution: The University of Sydney

Department: The Australian Centre for Microscopy and Microanalysis

Mailing Address:

Australian Centre for Microscopy & Microanalysis  
Madsen Building F09  
The University of Sydney  
NSW 2006 Australia

Phone: +61 2 9351 2351

Fax: + 61 2 9351 7682

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**Abstract:**

Advanced atom probe tomography (APT) techniques have been developed and applied to the atomic-scale characterisation of multi-component nano-structured materials with particular emphasis on the Ni-based superalloys with Platinum Group Metal (PGM) additions. AOARD Grant 094114 has enabled the development of entirely new analysis approaches for solute distribution/segregation analysis, atom probe crystallography, and lattice rectification. It has been demonstrated that these tools have potential for converging atomic scale modeling and simulation with atomic resolution microscopy. Our developments now sit on the cusp of a renaissance approach in materials design, where integrated computational materials engineering (ICME) can be enabled by real world 3D atomic resolution data via atom probe microscopy.

**Introduction:**

The ultimate goal of this ambitious study was to explore a highly topical question – *Given the instrumental and analytical limitations of atom probe tomography (APT), can we recreate a complete atomic system that is highly representative of the original specimen? Moreover, can we deploy such techniques to explore the precise solute architecture within key engineering solid solutions such as exist in PGM superalloys?*

Although APT is arguably the most powerful 3D atomic microscopy technique currently available – allowing tens of millions of atoms together with their chemical identity to be reconstructed, some limitations prevent perfect lattice resolution. These are primarily: a limited detector efficiency (typically less than 57%) as well as a spatial resolution just slightly above what is required to directly observe the site occupancy of individual atoms. However, a wealth of

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14. ABSTRACT <b>Advanced Atom Probe Tomography (APT) techniques have been developed and applied to the atomic-scale characterization of multi-component nanostructured materials with particular emphasis on the Ni-based superalloys with Platinum Group Metal (PGM) additions. This research has enabled entirely new analysis approaches for solute distribution/segregation analysis, atom probe crystallography, and lattice rectification and has demonstrated potential for converging atomic scale modeling and simulation with atomic resolution microscopy. These findings now sit on the cusp of a renaissance approach in materials design, where Integrated Computational Materials engineering (ICME) can be enabled by real world 3D atomic resolution data via atom probe microscopy.</b>					
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crystallographic information is commonly contained within APT reconstructions that could be used to restore the crystal lattice. The positions of the missing atoms within the reconstruction would then become apparent and could be replaced with the same measured short-range order as the detected atoms in the reconstruction to give a complete crystal model of the original specimen.

Such information would be invaluable to the study of structure-property relationships of nano-structured materials including the Ni-base superalloys. Most significantly, the simulated data derived from APT reconstructions could be incorporated directly into other analyses, e.g. molecular dynamics (MD) simulations and density functional theory (DFT) calculations.

A series of techniques have been developed at the University of Sydney to turn these concepts into reality and together represent a significant improvement on the current state-of-the-art in atom probe analysis. These techniques make up the specific aims of this research project:

- Characterise solute distribution/segregation
- Analyse the crystallographic information contained within APT reconstructions – determine crystallographic orientation
- Develop techniques to restore the atomic site occupancy within APT reconstructions based on observed crystallography – a process known as ‘lattice rectification’
- Develop a method to accurately simulate the missing atoms within atom probe reconstructions

## **Experiment/theory:**

### **Atom Probe Tomography**

APT is a powerful microscopy technique where by ions are successively field evaporated from the end of a needle-like specimen (typically with an end radius < 50 nm) towards a position sensitive detector. A schematic of the process is shown in figure 1(a). A back-projection model is then used to determine the original  $-x$ ,  $-y$  and  $-z$  co-ordinates of the ions. The time-of flight is also recorded, allowing for the mass-charge ratio (and subsequently the chemical identity) of each ion to be determined. The experiments were done under ultra-high vacuum using a DC standing voltage and either a high voltage or laser pulse to achieve field evaporation. A CAMECA Local Electrode Atom Probe (LEAP) 3000X and 3000X-Si (figure 1(b)) were used to collect experimental data in this project. Data was reconstructed on the commercially available IVAS software.

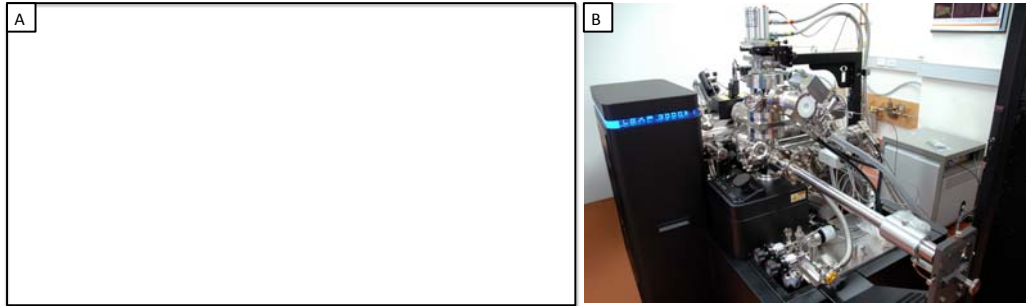


Figure 1. (a) The basic experimental operation of APT. (b) The LEAP 300X-Si at the University of Sydney.

### **Data Analysis Techniques**

A range of advanced APT analytical techniques have been developed and applied to the study of Ni-based superalloys and other multi-component materials to address the aims of the research project:

#### *Binomial frequency distribution analysis:*

The reconstruction is separated into blocks of a chosen amount of atoms, counting the number of a particular species of atom in each block and histogram the results. The results can be compared to a binomial distribution – the expected distribution of solute atoms assuming they are randomly distributed throughout the solid. Any shift in solute distribution away from the binomial can be attributed to atomic segregation within the reconstruction and can be quantified with statistical studies such as a  $\chi^2$  test.

#### *1D Spatial Distribution Maps (SDMs):*

1D SDMs histogram the inter-atomic distances within a volume of interest along a specified directional component. In regions surrounding poles (lower density regions corresponding to where a crystallographic direction intersects with the surface of the tip) a 1D SDM will often show a sinusoidal like distribution where the peaks represent atomic planes. If the reconstruction is then rotated slightly around the -x and then -y axis by  $\theta^\circ$  and  $\phi^\circ$ , the signal to noise ratio of the SDM along a particular direction will change – the maxima corresponding to the crystallographic direction relative to the detector.

If three non-collinear poles within the reconstruction can be characterized in this way it is then possible to determine the orientation of the tip relative to the detector face. For more information see Moody et al. [1].

#### *Lattice Rectification:*

Lattice rectification is a powerful and relatively new analysis technique that has been developed largely in part at the University of Sydney and was advanced further throughout the project. The process involves restoring the original site occupancy of atoms within the experimental reconstruction (shown in Figure 2 (a)). Several methods to do this have been proposed over the past decade but this research primarily involves using ‘real space’ (as apposed to reciprocal space) SDM techniques to determine crystal structure and restore the lattice based on this information. For more detail see Moody et al. [1].

Other methods were developed and trialed throughout the research for reasons highlighted in the results section of this report. This includes a method that minimises the difference between the experimentally reconstructed inter-atomic distances and that of the theoretically known lattice. Once a reconstruction is rectified the site occupancy of different species of atoms can be characterized. For more detail see figure 10.

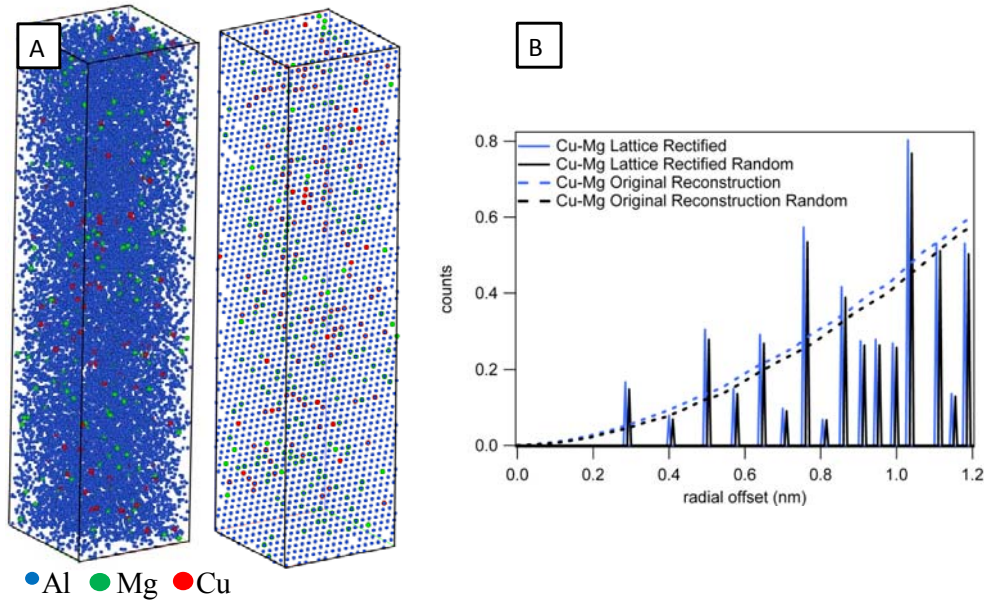


Figure 2 (a) A subvolume of an Al-Mg-Cu alloy before lattice rectification (left) and after lattice rectification (right). (b) Partial RDFs before and after rectification

#### Radial Distribution Functions (RDFs):

RDFs histogram the radial inter-atomic distances within a volume of interest. A partial-RDF only considers atoms of a particular atomic species. An example of partial RDF's is shown in figure 2 (b). This analysis technique is useful for confirming crystal structure, detector efficiency and solute segregation in lattice-rectified reconstructions.

#### Simulating missing atoms:

Lattice rectification also has the advantage of enabling the missing atoms in a reconstruction to be identified. A procedure has been designed at the University of Sydney to do this and forms part of this research project - shown in figure 3.

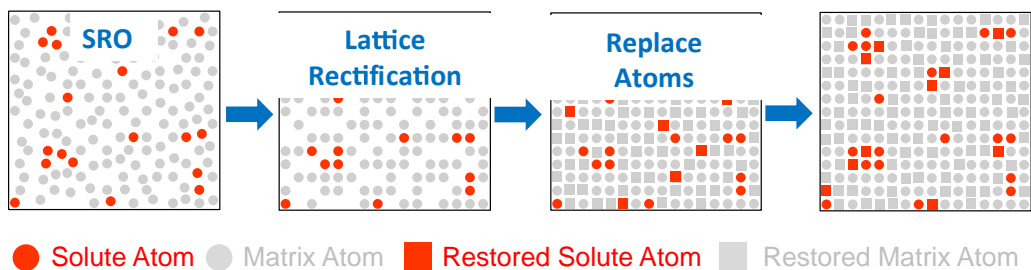


Figure 3 – procedure for simulating the missing atoms in an APT reconstruction

In the first step, the short-range order (SRO) is measured within the experimental reconstruction – this is essentially a measure of the degree at which atoms of different species have segregated towards or away from each other. For multi-component materials, such as the Ni-based superalloys, a generalised multi-component short-range order (GM-SRO) parameter has been developed at the University of Sydney by Ceguerra et al. [2] to accurately describe segregation phenomena between many different atomic species. It was recently demonstrated that SRO remains constant for detector efficiencies greater than 50 % meaning that measurements obtained from APT experiments can reflect the true SRO of the system. The SRO in a perfect lattice and an imperfect experimental APT reconstruction are shown in figure 4 (a) and (b), the GM-SRO formula is shown in (c).

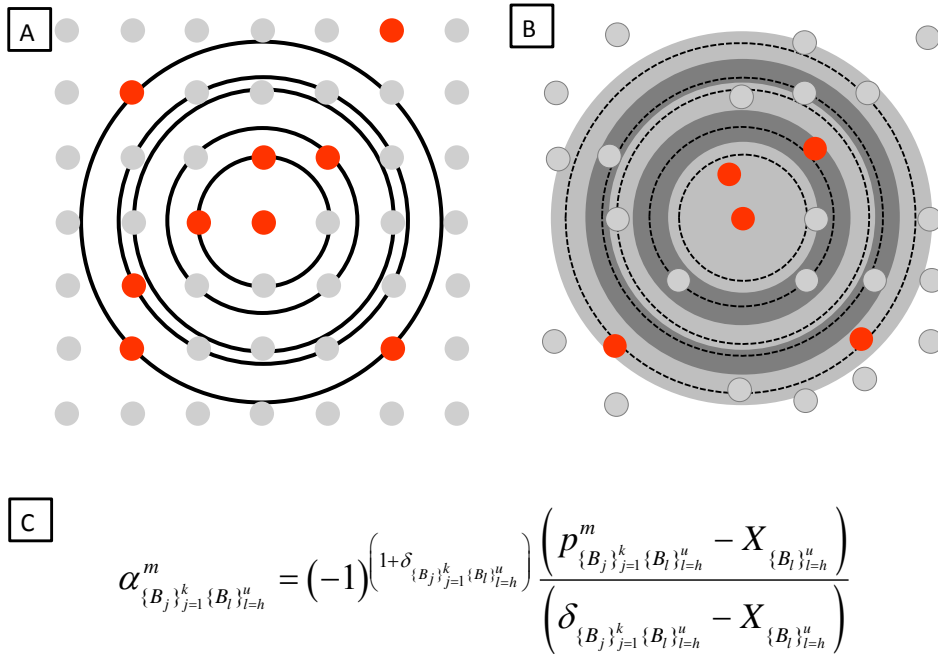


Figure 4 (a) SRO in a perfect lattice. (b) SRO in an imperfect lattice. (c) The GM-SRO formula [2]

In the next step, the experimental reconstruction is rectified. Atoms are missing in the lattice due to the limited detector efficiency of APT (figure 3 step 2).

An algorithm that determines the sites of the missing atoms within the boundary of the reconstruction is then used (figure 3 step 3) – the design of which was part of this research project. Each atom can then be assigned an element to match the measured composition in the experimental reconstruction. In a final step (figure 3 step 4) – a monte-carlo simulation can be used to move the positions of these replaced atoms towards the measured GM-SRO. A complete crystal model of the specimen is then created from the rectified originally detected atoms and the replaced atoms

## Results and Discussion:

An example of experimental data collected from a Ni-base superalloy with Platinum Group Metal (PGM) additions is shown in figure 5. The data was collected at 20 K using a 90 mm flight path and 25% pulse fraction and was provided by Van Sluytman et al. [3]. A selection of results from this data addressing aim 1 of the research project has been provided. If a thin slice of  $\sim 1.5$  million atoms (volume within blue cylinder in figure 5(a)) is clipped from the reconstruction and each atomic species is observed along the  $-z$  direction relative to the detector (figure 5(b)) it becomes visually apparent that certain elements have segregated to different regions. Al, Ta, W and Hf are all shown to segregate to the left and is a strong indication of the  $\gamma'$  phase. Cr is observed to do the opposite – partitioning more to the  $\gamma$  phase. No visual segregation is observed for Pt. Figure 5(c) shows the corresponding mass spectrum – clearly indicating the elements present within the data – providing valuable compositional information of the material.

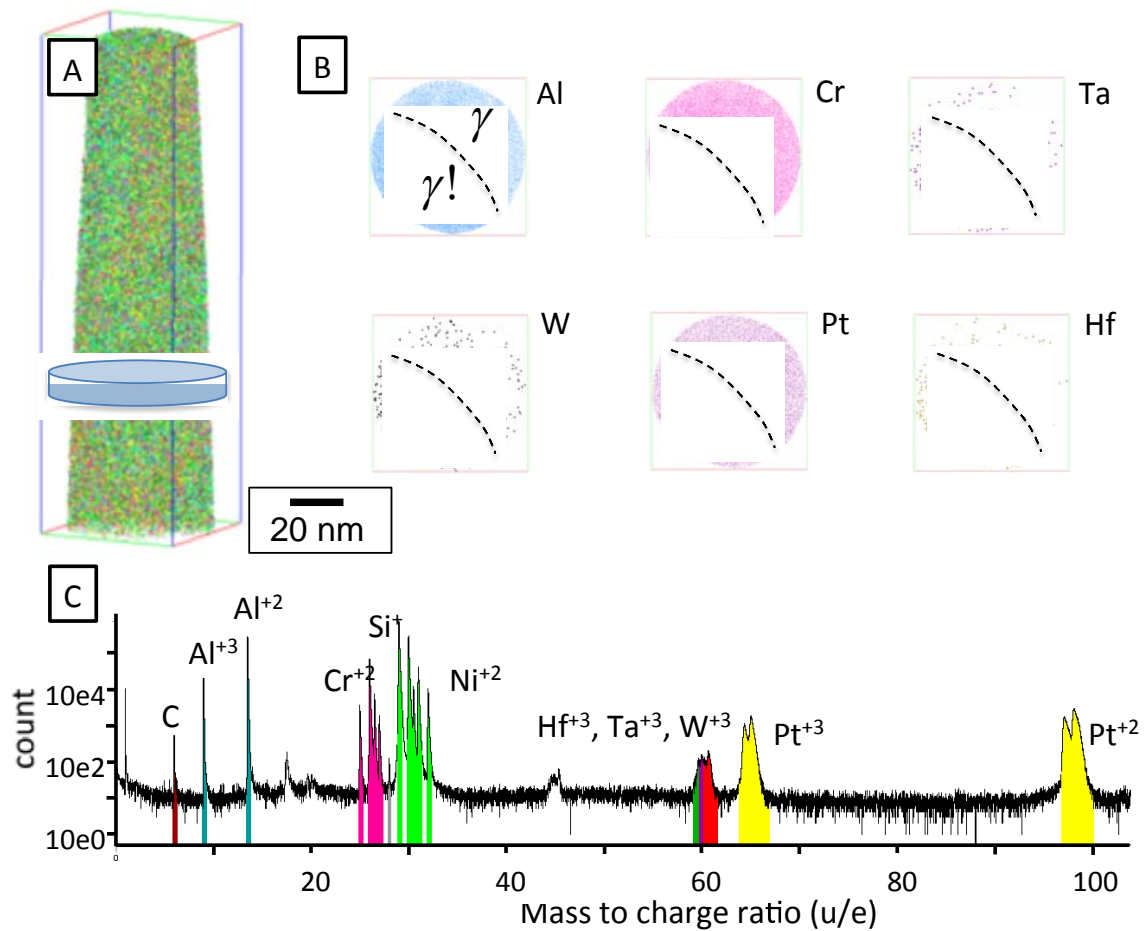


Figure 5. (a) APT reconstruction of a Ni-based superalloy with PGM additions – a thin blue cylinder (containing  $\sim 1.5$  million atoms) shows the subvolume extracted for further analysis. (b) Different elements within subvolume showing the segregation to the gamma and gamma prime phases. (c) Corresponding mass spectrum with ranged elements. Data courtesy Van Sluytman et al. [3]

For a more quantitative study on solute partitioning there are a number of data mining tools available such as a binomial frequency distribution analysis that is used in figure 3 to compare the experimental Al distribution to a completely random distribution. Al is shown to have a bi-modal distribution consistent with the  $\gamma/\gamma'$  partitioning observed in figure 6(b). A contingency table analysis [4] and a clustering analysis such as the core-linkage algorithm [5] developed at the University of Sydney can also be helpful in further determining nano-scale segregation phenomena.

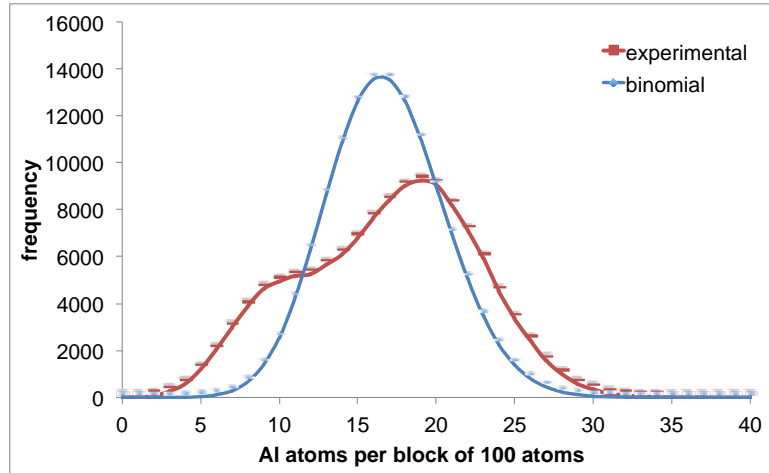


Figure 6. Al frequency distribution. Al concentration is 16.83 at. %, block size is 100 atoms. The distribution of experimental Al atoms is significantly different to a completely random binomial distribution.

Aim 2 of the research project was achieved primarily through the use of SDMs to observe crystallographic information. Figure 7 shows a density map of the same volume used in figure 2. The dark blue regions are known as poles and can be indexed to certain crystallographic orientations as shown – similar to that of a stereographic projection. 1D SDMs were then created from cylindrical regions of interest 4 nm in diameter around three non-collinear poles and are shown in figure 4(b). Signal corresponding to crystallographic planes is evident. The rotations around the  $-x$  and  $-y$  axis to achieve the highest signal to noise ratio are also given and were used to determine the crystallographic orientation of the tip relative to the detector.



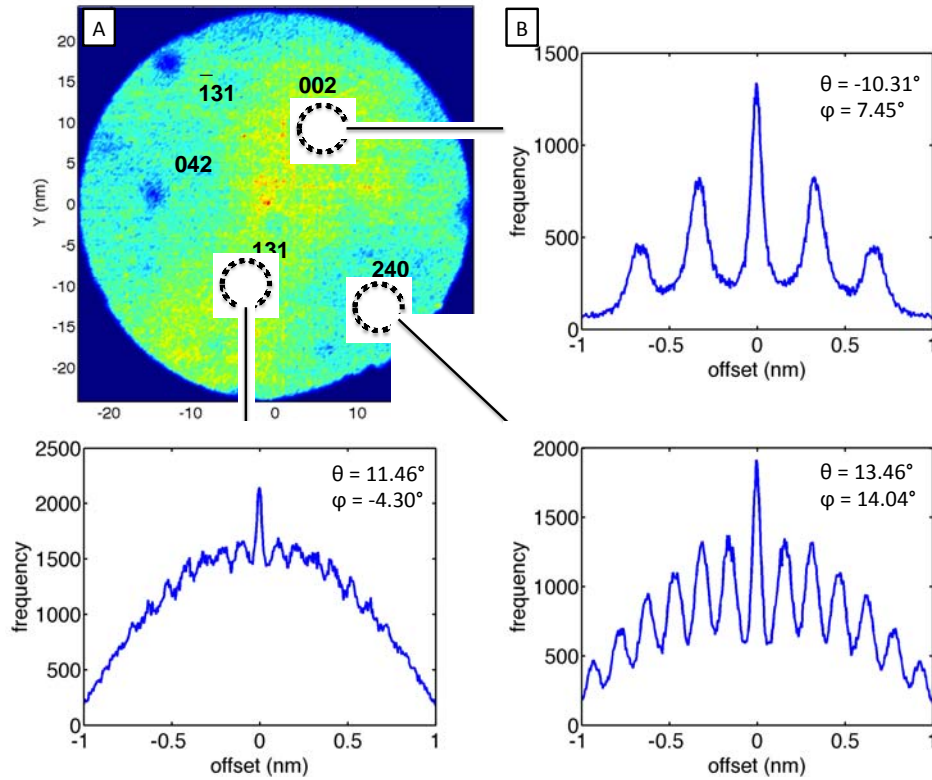


Figure 7. (a) A 2D density map of the same subvolume shown in figure 2 with indexed poles. (b) 1D SDMs created within volumes surrounding 3 non-collinear poles – the rotation around the x and y axis to give the highest signal to noise ratio i.e. the crystallographic orientation relative to the detector is also given.

The ordered structure of the  $\gamma'$  phase could also be clearly observed within the reconstruction. Figure 8 (a) shows the Al (blue) and Ni (green) on (002) planes within the standard reconstruction as well as a species specific 1D SDM in the [002] direction – clearly revealing how Al occupies every alternate plane. The amplitude of the Al peaks is almost the same as the alternate Ni peaks suggesting that in every alternate (002) plane Al and Ni exist in almost equal quantities. This agrees extremely well with the theoretically known ordered  $L1_2$  crystal structure of  $\gamma'$ . Although ladder diagrams have shown similar information in 1D atom probe data – this represents the first time that SDMs have been used to so elegantly show this ordered structure in a 3D atom probe reconstruction. It is a testament of the spatial integrity of the reconstruction and suggests that identifying the site occupancy of individual atoms is a realistic possibility.

The 3<sup>rd</sup> aim of the research project was then addressed by applying lattice rectification techniques to restore the crystal structure of the specimen. Figure 8 (b) shows the same volume after lattice rectification using the protocol described by Moody et al[1]. Almost all Al atoms have been positioned correctly on alternate (002) planes and ordered structure becomes strikingly apparent.

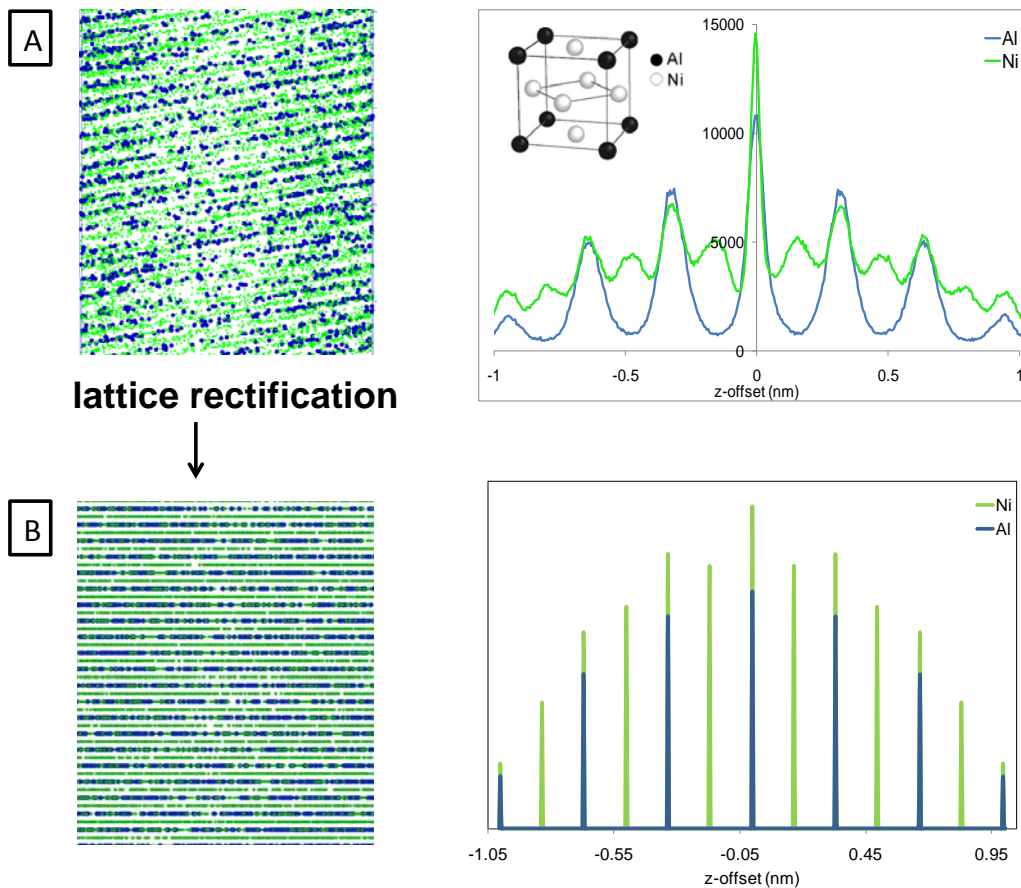


Figure 8. (a) Subvolume of Ni-based superalloy in gamma prime along [002] zone axis as well as species specific 1D SDMs showing the plane occupancy of Al and Ni (b) the same volume after lattice rectification

Figure 9 (a) shows the same region of interest as figure 8 except that Al (blue) and Cr (red) atoms are now shown. The Cr atoms are observed to occupy the same (002) planes as the Al atoms – direct evidence that Cr substitutes for Al and not Ni in the  $\gamma'$  lattice. Figure 9 (b) further confirms the accuracy of the lattice rectification algorithm developed – perfectly restoring Al and Cr onto the same discrete (002) planes.

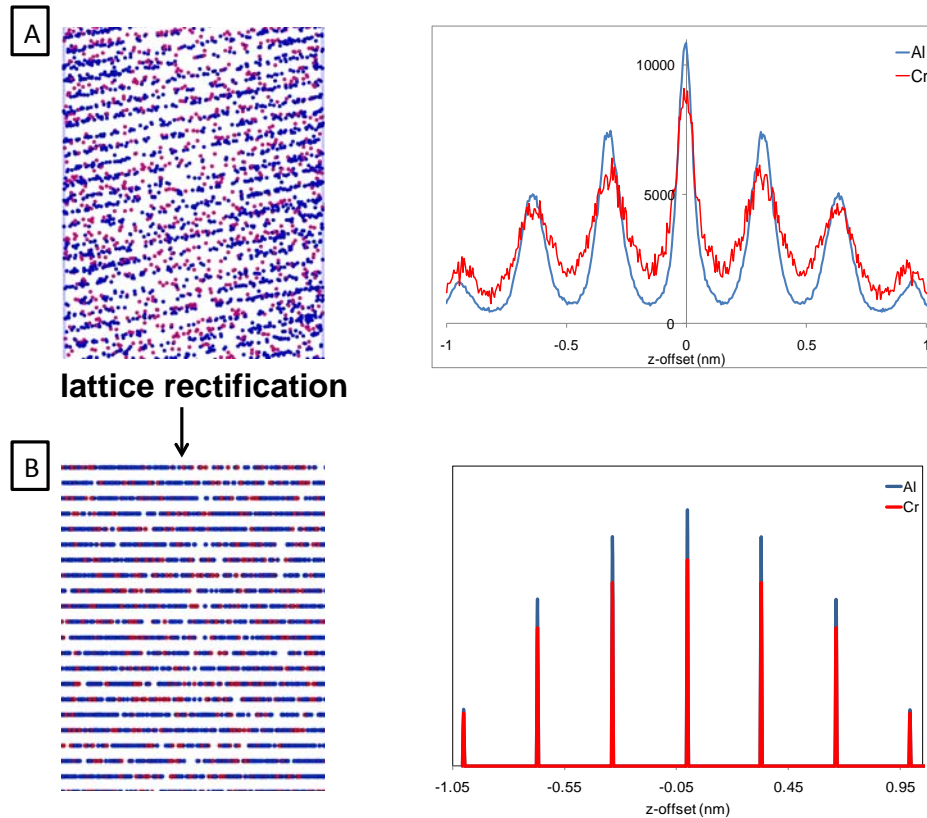


Figure 9. (a) Subvolume of Ni-based superalloy in  $\gamma'$  phase along  $[002]$  zone axis as well as species specific 1D SDMs showing the plane occupancy of Al and Cr (b) the same volume after lattice rectification

Although lattice rectification was already producing impressive results, some issues were apparent:

- Could not rectify non-bravais lattices such as diamond cubic
- Crystal orientation and structure needed to be confirmed in experimental reconstruction
- Difficulties with reference atoms and separating atoms that were rectified to the same lattice site (multiple occupancies)

The 3<sup>rd</sup> aim of the research project was further addressed by developing more sophisticated lattice rectification techniques that could be more easily implemented by the user to different crystal structures and to a higher degree of accuracy. Figure 10 (a) shows the major steps of the most current algorithm developed:

- 1) The inter-atomic distances within the experimental reconstruction are measured.
- 2) An atom is randomly selected from the experimental reconstruction and placed at the origin of a perfect lattice.
- 3) The next nearest atom within the experimental reconstruction is positioned on the perfect lattice to minimise the difference from the measured inter-atomic distance.
- 4) The next closest atom within the experimental reconstruction to those that have been rectified is then positioned on the lattice to minimise the difference from measured inter-atomic distances. This is repeated for all remaining atoms.

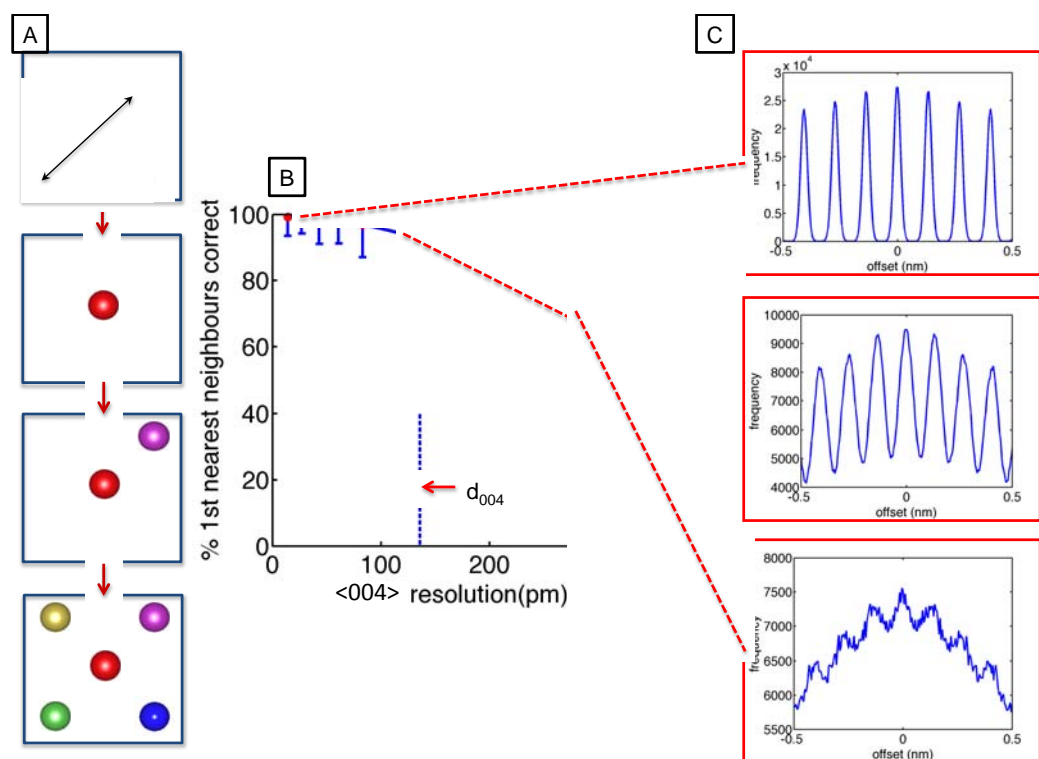


Figure 10 – (a) major steps in new lattice rectification method (b) the % of 1<sup>st</sup> nearest neighbouring atoms correct after simulated Si datasets of 1000 atoms with worsening spatial resolution were rectified. (c) [004] 1D SDMs showing measurable plane structure in simulated datasets

The accuracy of this method was then tested using Matlab software. A simulated Si dataset (with diamond cubic crystal structure) containing 1000 atoms had different levels of noise applied in -x, -y and -z – including levels similar to that observed in experimental Si atom probe data ~ 350 pm. The % of first nearest neighbouring atoms restored after rectification for different starting atoms was then compared (figure 10 (b)). Even for reconstructions with measured spatial resolution similar to experimental Si, ~ 65 % of 1<sup>st</sup> nearest neighbouring atoms were restored on average but this was measured as high as 92 % depending on the atom chosen for the rectification procedure. Most atoms that were not positioned on exactly the right lattice site were only one lattice site away. Figure 10 (c) shows [004] 1D SDMs for different levels of spatial resolution. Methods for better selecting a reference atom continue to be worked on. The method has the benefits of being applicable to any crystal structure without the need to characterise the crystallographic orientation before hand.

Figure 11 shows this new lattice rectification technique being applied to experimental Sb doped Si atom probe data. Data was collected using voltage pulsing (25% pulse fraction) at 25 K with a 90 mm flight path. Figure 11 (a) shows the resulting reconstruction containing ~ 6 million atoms (Sb was measured to be ~ 0.06 at. %). Figure 11 (b) shows a small subvolume from the reconstruction. Figure 11(c) shows this same volume after lattice rectification – clearly revealing crystal structure and the likely site occupancy of the dopant Sb atoms.

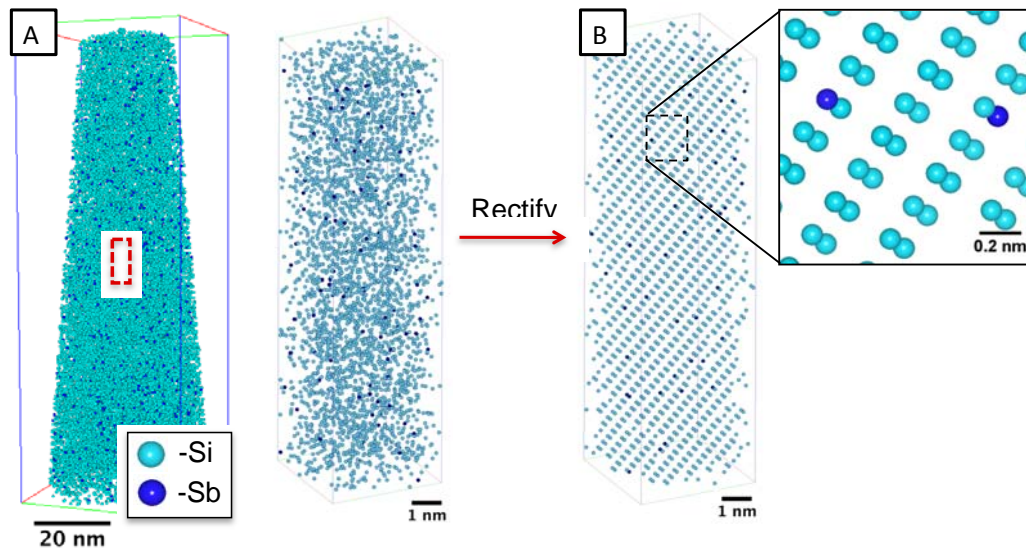


Figure 11. (a) Experimental Sb doped Si reconstruction. (b) Subvolume from reconstruction (c) Sum-volume after rectification showing site occupancy of dopant atoms

An example of results concerning the final aim of the research project is shown in figure 12 that demonstrates how a rectified reconstruction can be used to reveal missing atoms – these atoms can then be restored with the same measured short range order as those that were detected. Once combined, a complete crystal model of the material is possible which can then be used in sophisticated MD and DFT simulations to elucidate structure property relationships.

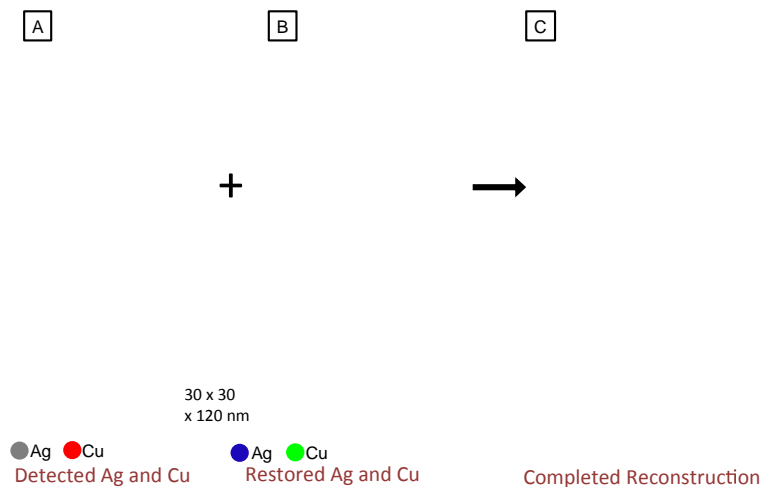


Figure 12 – (a) A rectified experimental APT reconstruction of an Ag and Cu alloy (b) the missing atoms of the rectified reconstruction with restored composition and short range order (c) The two reconstructions combine to give a complete crystal model of the material

## Summary, Conclusions and Next Steps:

In summary, this work offers a significant contribution to high-end atom probe analysis of multi-component materials – particularly that of Ni based superalloys with PGM additions. A variety of analysis techniques have been developed and applied throughout the project – with an emphasis on studying the solute distributions and crystallography present in atom probe reconstructions.

We conclude that these analysis tools are a plausible route to enable ICME approaches to materials design and development. Our lattice rectification methodology is an especially important step towards the ultimate microscopy – that of imaging the exact 3D atomic architecture of nano-structured materials.

The next steps, should such an opportunity present itself, would be for a larger project geared around a specific materials design challenge where a target range of materials properties and performance are identified and the team from Sydney are more tightly integrated into the materials development teams in (e.g.) the USA. We propose that a three-tiered collaboration with capacity in (i) materials processing, (ii) multi-scale materials modeling, and (iii) multi-scale materials collaboration be established.

#### **List of Publications and Significant Collaborations that resulted from your AOARD supported project:**

##### **Papers published in peer-reviewed journals:**

- M.P. Moody, B. Gault, L.T. Stephenson, R.K.W. Marceau, R.C. Powles, A.V. Ceguerra, A.J. Breen, S.P. Ringer, *Lattice Rectification in Atom Probe Tomography: Toward True Three-Dimensional Atomic Microscopy*, Microscopy and Microanalysis, 17 (2011) 226-239
- Kelvin Y. Xie, Andrew J. Breen, Lan Yao, Michael P. Moody, Baptiste Gault, Julie M. Cairney, Simon P. Ringer, *Overcoming Challenges in the study of nitrided microalloyed steels using atom probe*, Ultramicroscopy, 112 (2012) 32-38

##### **Manuscripts Submitted but not yet published/ manuscripts in preparation:**

- Andrew J. Breen, Michael P. Moody, Baptiste Gault, Anna V. Ceguerra, Kelvin Y. Xie, Sichao Du, Simon P. Ringer, *Spatial Decomposition of Molecular Ions Within 3D Atom Probe Reconstructions* (submitted to Ultramicroscopy)
- Andrew J. Breen, Kelvin Y. Xie, Michael P. Moody, Julie M. Cairney, Simon P. Ringer, *Resolving the Orientation of Ultra-Fine High Field Precipitates* (in preparation for Scripta Materialia)
- Andrew J. Breen, Michael P. Moody, Leigh T. Stephenson and Simon P. Ringer, *Restoration of the Diamond Cubic Lattice in a Silicon-Based APT Reconstruction* (in preparation for Microscopy and Microanalysis)
- Michael P. Moody, Anna V. Ceguerra, Andrew J. Breen, Baptiste Gault, Xiang Yuan Cui,



Leigh Stephenson, Ross K. W. Marceau, Rebecca Powles, Simon P. Ringer, *Completing the Picture – Enhanced Atom Probe for Complete 3D Atomic Imaging and Simulation* (in preparation for Nature Communications)

**Conference presentations:**

- Andrew J. Breen, Michael P. Moody, Leigh T. Stephenson, Simon P. Ringer, *Restoration of the Diamond Cubic Lattice in a Silicon-Based APT Reconstruction*, 52<sup>nd</sup> International Field Emission Symposium (IFES), July 2010 (poster presentation)
- Andrew J. Breen, Michael P. Moody, Leigh T. Stephenson, Simon P. Ringer, *3D Imaging of Lattice Structure in Silicon using Atom Probe Tomography*, 7<sup>th</sup> Pacific Rim International Conference on Advanced Materials and Processing (PRICM7), August 2010 (Oral Presentation)
- Andrew J. Breen, Michael P. Moody, Baptiste Gault, Anna V. Ceguerra, Leigh T. Stephenson and Simon P. Ringer, *Towards Spatial Decomposition of Complex Ions*, 1<sup>st</sup> Cameca Atom Probe User Meeting, May 2011 (Oral Presentation)
- A.J. Breen, K.Y. Xie, M.P. Moody, J.M. Cairney, S.P. Ringer, *Resolving the morphology and orientation of ultra-fine high field precipitates*, 53<sup>rd</sup> International Field Emission Symposium (IFES), May 2012 (oral presentation)

**Interactions with industry or with Air Force Research Laboratory scientists or significant collaborations that resulted from this work:**

- Research visit to Iowa State University in February 2010. Discussions and collaborative work on data-mining and crystallography analysis techniques on multi-component materials including Ni-based superalloys. Discussions and collaborative effort is ongoing.
- Research visit to National Institute of Materials Science (NIMS), Tsukuba Japan, November 2011. Discussions were had with Prof. Hiroshi Harada and the High Temperature Materials group about collaborative research on a single crystal Ni-based superalloy for fundamental APT research purposes – negotiations on an alloy to collect data on in Sydney are ongoing. Prof. Kazuhiro Hono and the Magnetic Materials Unit were also visited – collaborative work on the effect of laser spot-size and frequency on a selection of multi-component materials was done.

**References:**

- [1] M.P. Moody, B. Gault, L.T. Stephenson, R.K.W. Marceau, R.C. Powles, A.V. Ceguerra, A.J. Breen, S.P. Ringer, *Microscopy and Microanalysis*, **17** 226–239 (2011).
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